Emerging Scientific Opportunities Using X-ray Imaging at the APS

Wah-Keat Lee (APS, ANL)

Contributors:

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Y. Chu & F. DeCarlo - 2-BM

P. Zschack, C. Rau & G. Long - 33&34 ID

K. Fezzaa - 1-ID

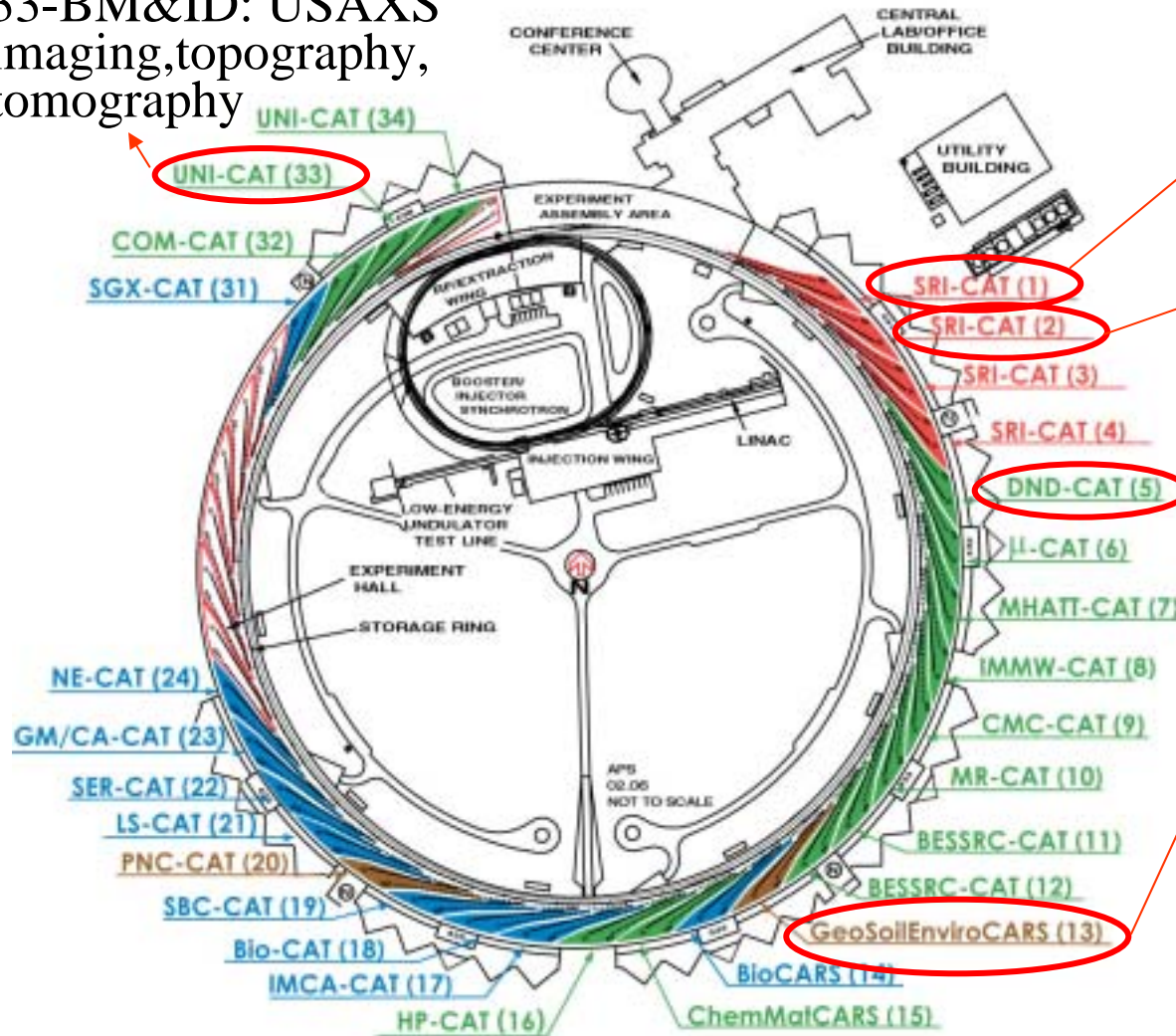**ADVANCED PHOTON SOURCE**

Workshop Focus

- Full field hard (> 5 keV) x-ray imaging

- Scientific applications - present & potential

- Cover broad and diverse disciplines

Outline:

- APS full-field imaging resources

- Selected science applications by technique

    - Topography

    - Topography & Fresnel diffraction

    - Tomography

    - USAXS imaging

    - Live phase-enhanced imaging

- Perspectives

33-BM&ID: USAXS imaging,topography, tomography

UNI-CAT (34)

UNI-CAT (33)

COM-CAT (32)

SGX-CAT (31)

CONFERENCE CENTER

CENTRAL LAB/OFFICE BUILDING

UTILITY BUILDING

EXPERIMENT ASSEMBLY AREA

RF/EXTRACTION WING

BOOSTER INJECTOR SYNCHROTRON

INJECTION WING

LINAC

LOW-ENERGY UNDULATOR TEST LINE

EXPERIMENT HALL

STORAGE RING

NE-CAT (24)

GM/CA-CAT (23)

SER-CAT (22)

LS-CAT (21)

PNC-CAT (20)

SBC-CAT (19)

Bio-CAT (18)

IMCA-CAT (17)

HP-CAT (16)

APS 02.06 NOT TO SCALE

N

SRI-CAT (1)

SRI-CAT (2)

SRI-CAT (3)

SRI-CAT (4)

DND-CAT (5)

μ-CAT (6)

MHATT-CAT (7)

IMMW-CAT (8)

CMC-CAT (9)

MR-CAT (10)

BESSRC-CAT (11)

BESSRC-CAT (12)

GeoSoilEnviroCARS (13)

BioCARS (14)

ChemMatCARS (15)

1-ID:Phase-enhanced live-radiography

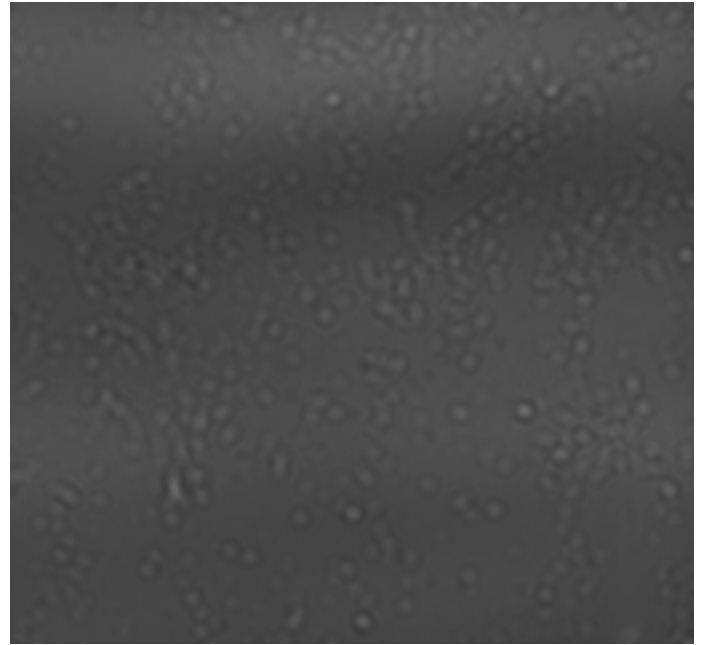2-BM:tomography, phase-enhanced topography

5-BM:tomography

13-BM:tomography

MATERIALS, CHEMICAL, & ATOMIC SCIENCE
BIOLOGY
GEO, SOIL, & ENVIRONMENTAL SCIENCE
INSTRUMENTATION

Only 2-BM is imaging > 50% of beamtime.

ADVANCED PHOTON SOURCE

No dedicated imaging beamline at the APS.
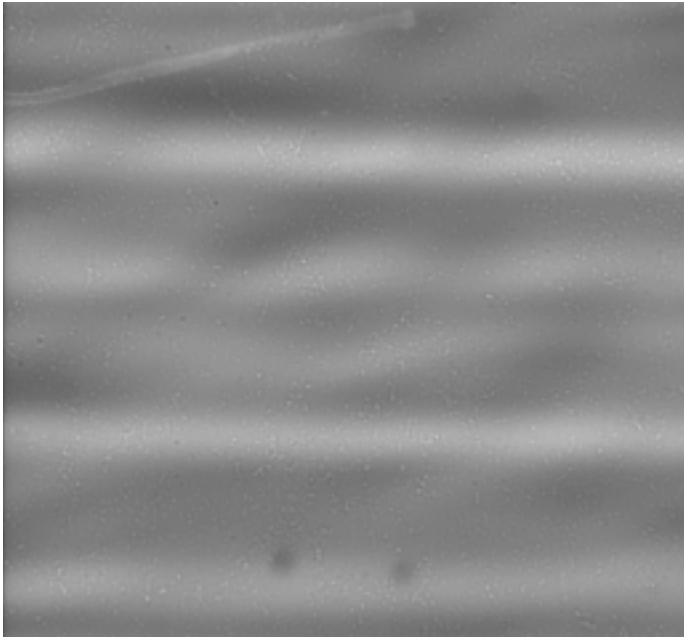
FOV ~ 1.3 mm

FOV ~ 0.3 mm

Beam artifacts and fluctuations complicate development.
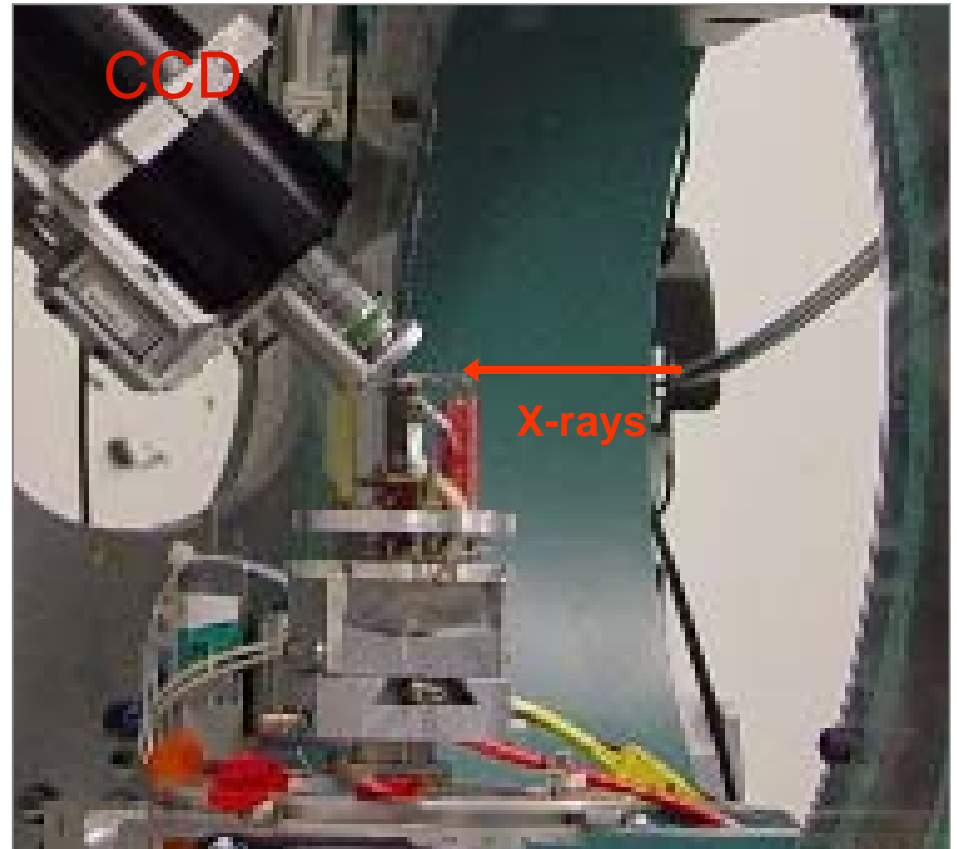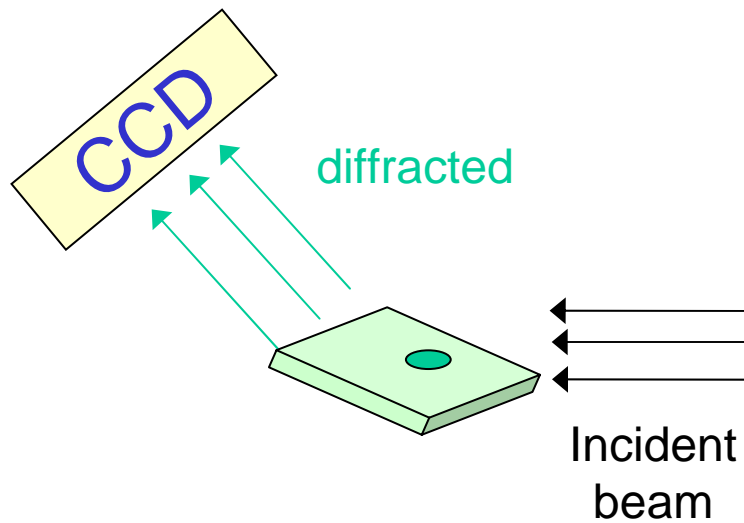
Microscope at UNICAT 34 ID

Undu-lator   Mirror   Mono   Sample   Condenser   Objective   2-D detector

53 m
53.1 m
0.45 m
0.35 m
0.025 m   0.5 m
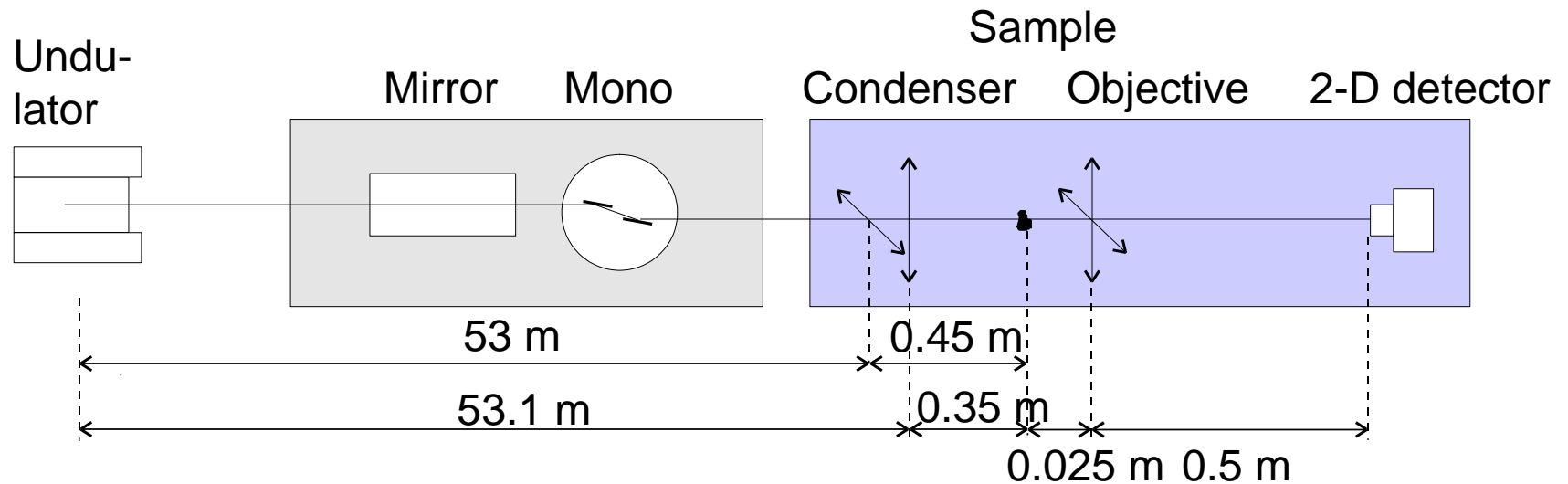
Resolution: 100 nm

Field of view: 100 micron

Energy: 6 - 13 keV

Status: Under commissioning

Christoph Rau & Paul Zschack

Topography

Full-Field X-ray Diffraction Imaging (topography) of Crystalline Samples

CCD

diffracted

Incident beam

CCD

X-rays

D-spacing of substrate, buffer and thin film are different -> depth-sensitive topographs
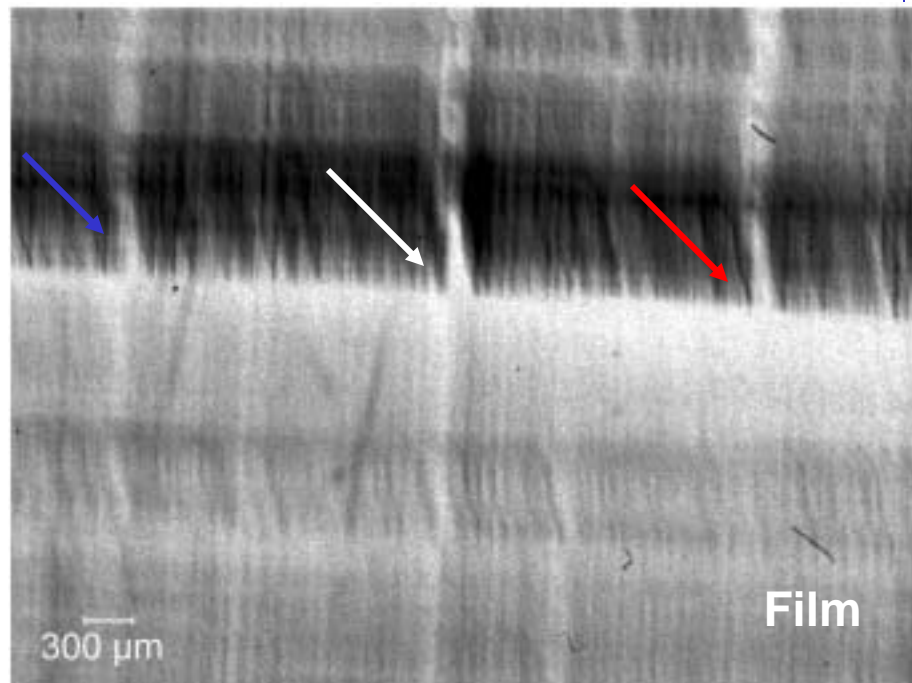
Si substrate
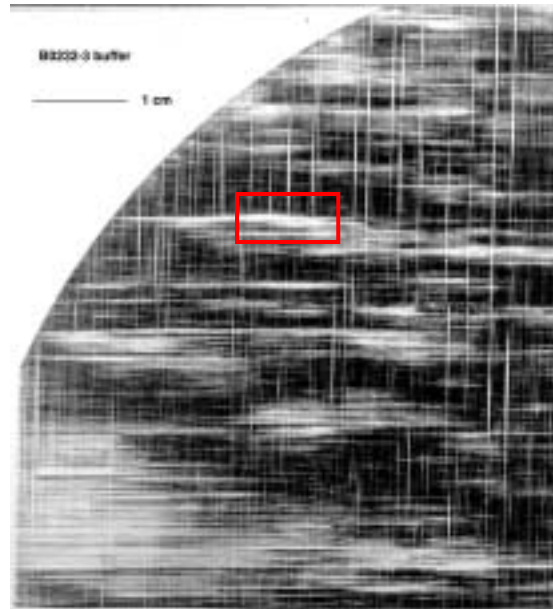
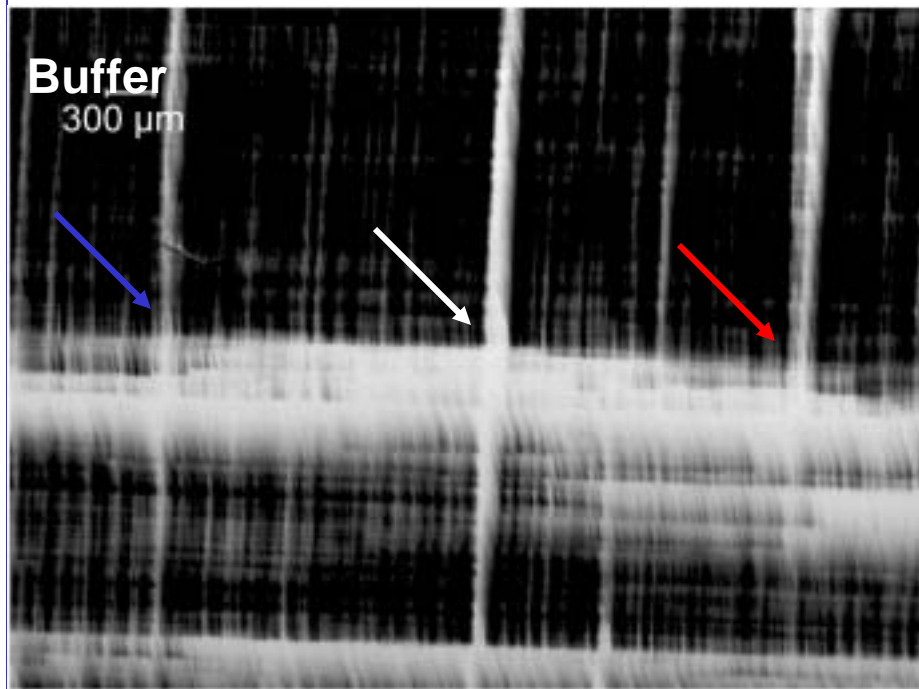Si/Ge buffer

500 Å strained Si film
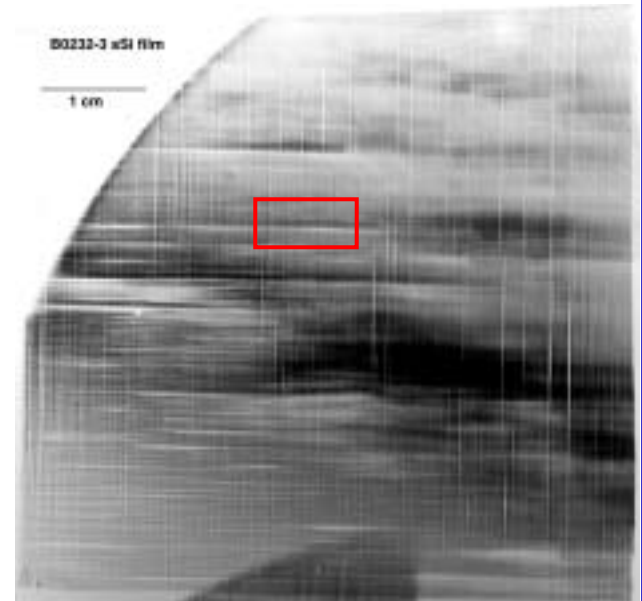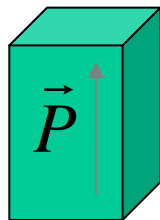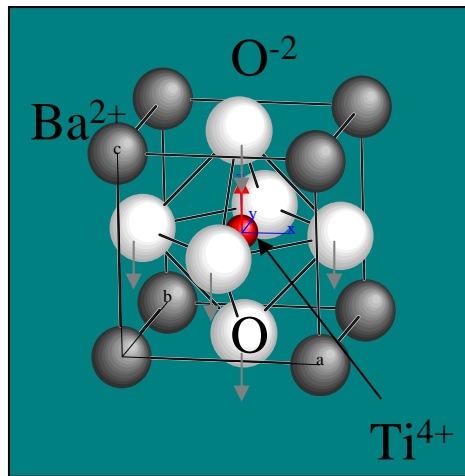
Si film
Si/Ge buffer
Si substrate
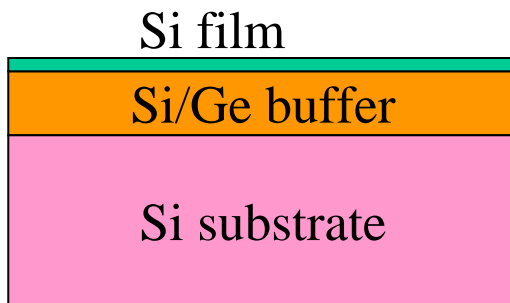
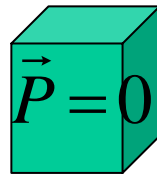Next generation Si based electronic materials

UNICAT

Strain propagates from buffer layer to thin film.
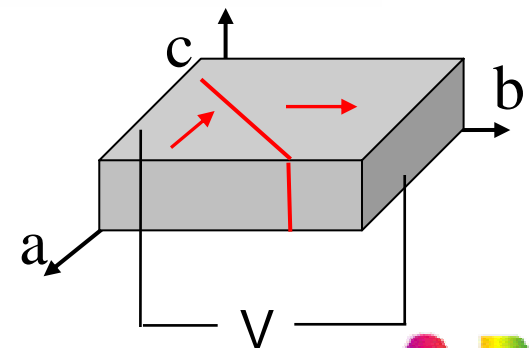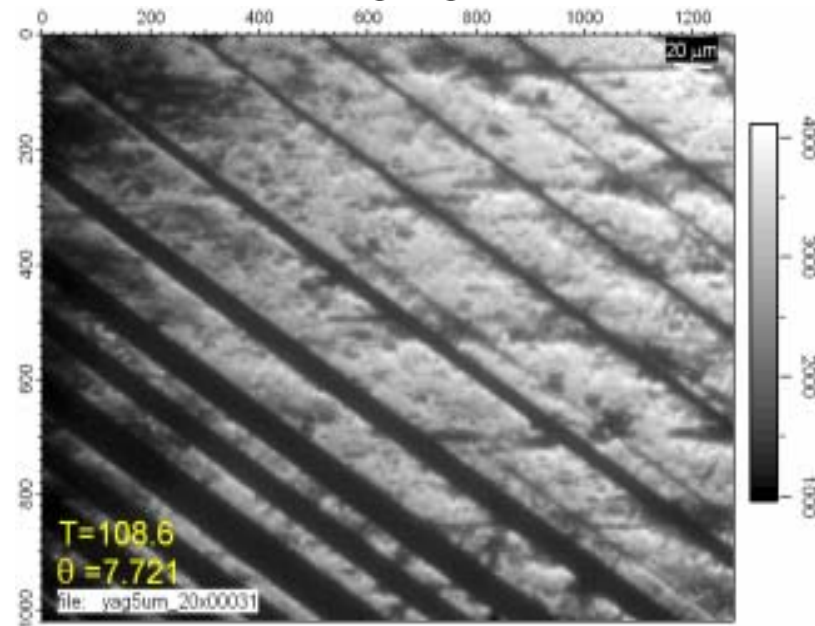
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Time resolved topography of ferroelectric Domains in BaTiO$_3$

- To study nucleation and growth of ferroelectric domains
- To develop time-resolved diffraction imaging technique

O^{-2}

Ba^{2+}

O

Ti^{4+}

$\vec{P}$ $\vec{P}=0$

Tetragonal   Cubic
T<120 °C   T>120 °C

T=108.6
θ =7.721
file: yag5um_20x00031
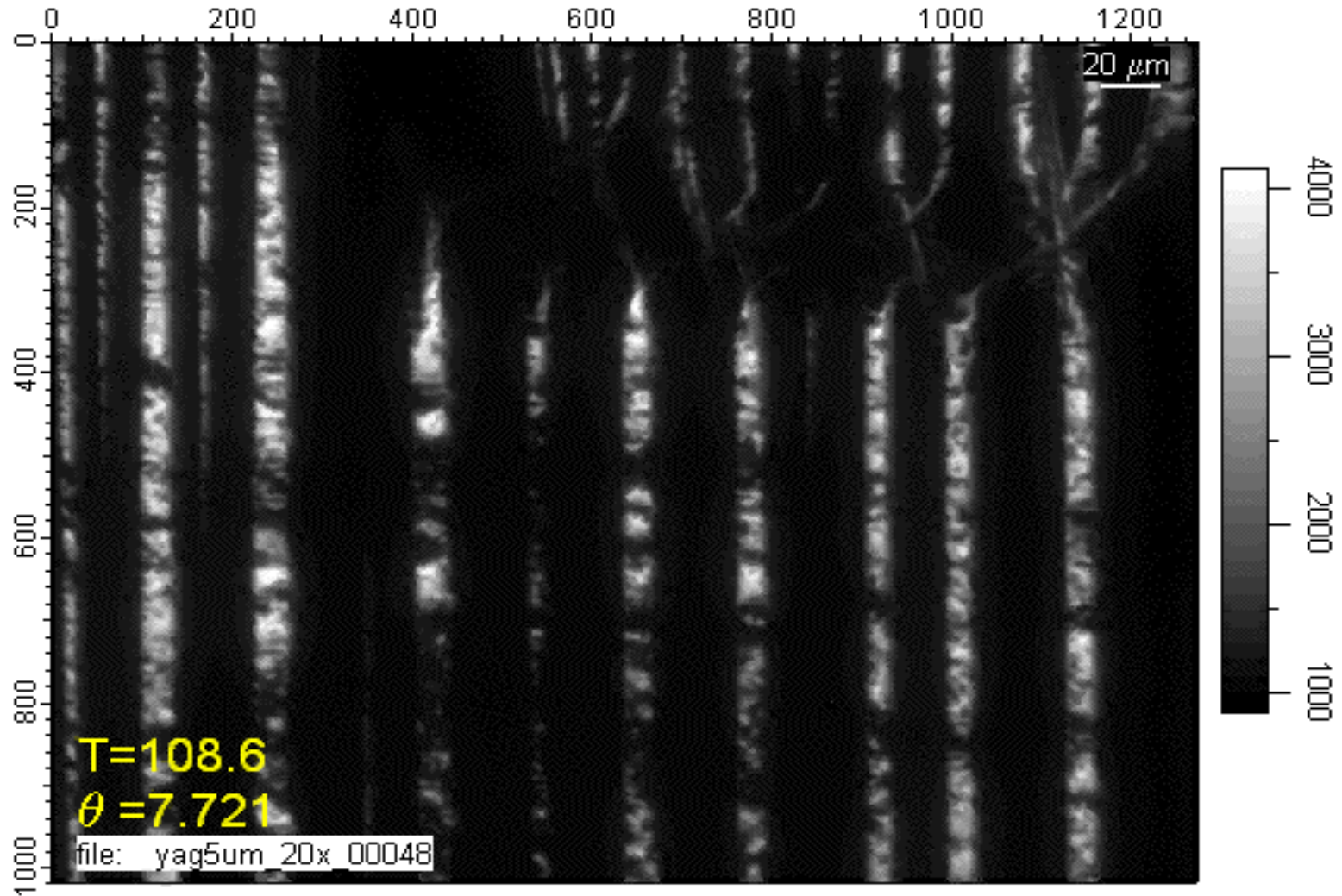

c   b

a   V

Tkachuk, Chu (XOR/ANL), You (MSD/ANL)

2-BM

Nucleation of 90° Ferroelectric Domains Induced by External Potential

At 108.6 °C with 2 keV/cm -> 0 keV/cm

See talk by Chu (C1)

Combine topography & Fresnel diffraction ……
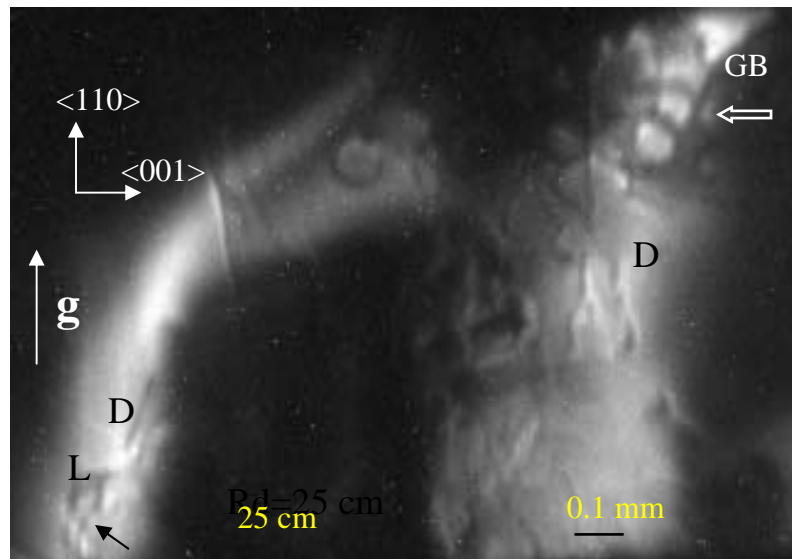
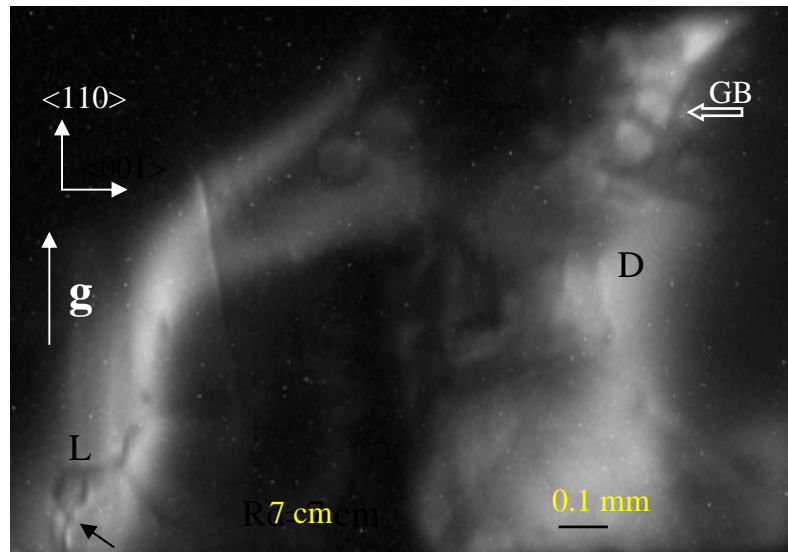# Diffraction Imaging from Biological Crystals

- Investigation of growth and impurity defect in protein crystals

- Defect-contrast enhancement from weakly diffracting biological crystals achieved using phase-contrast

(Topography & Fresnel diffraction)

high-resolution CCD system

R_d

partially coherent monochromatic x-rays

perturbed region

perfect region

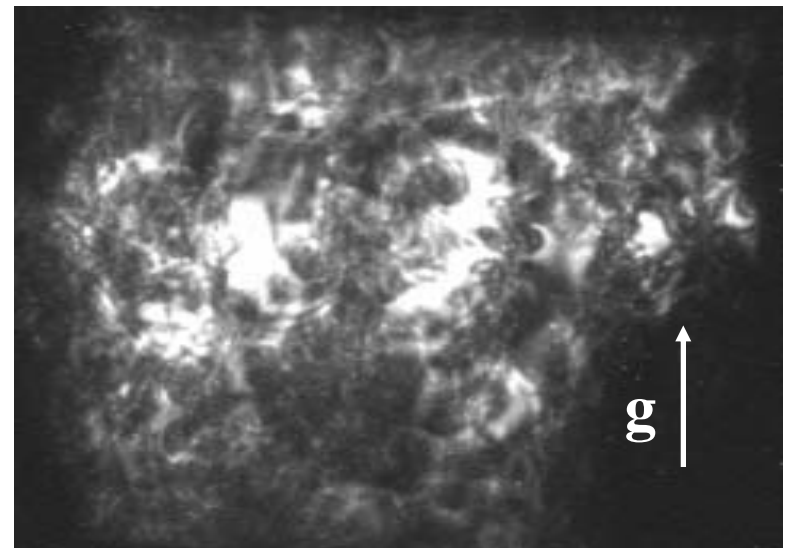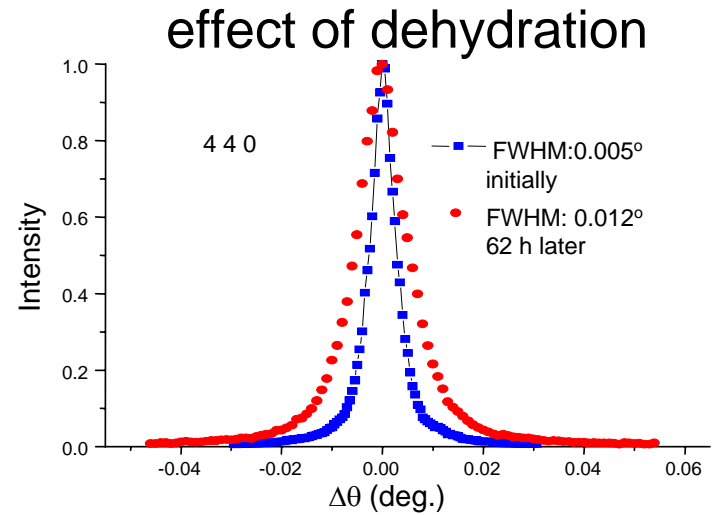Z. W. Hu, USRA, NASA/Marshall Space Flight Center
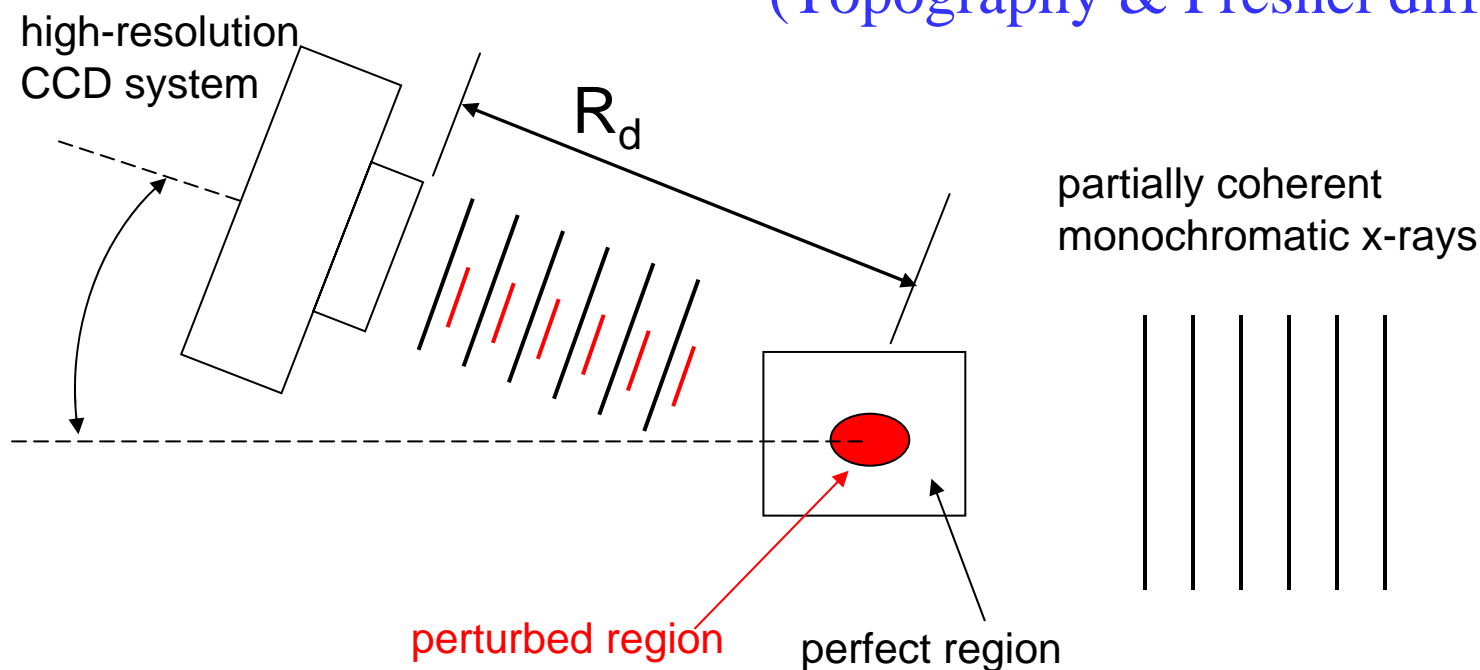
Phase-Contrast Topography of Lysozyme Crystals
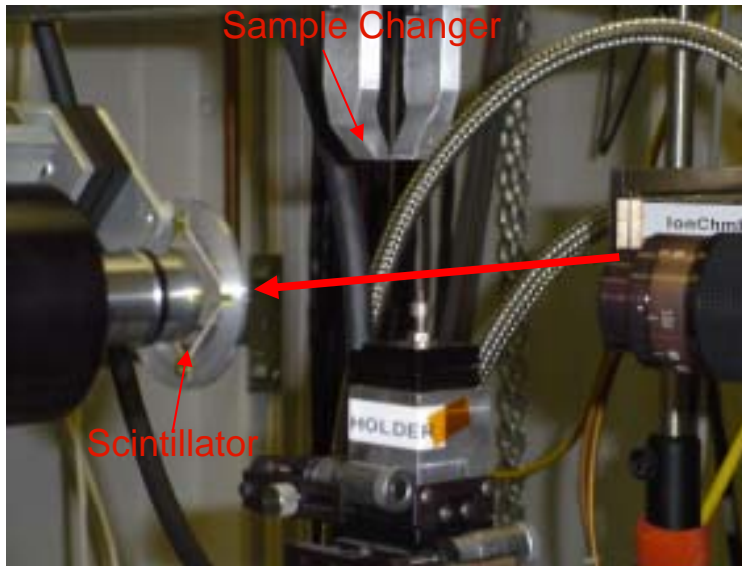

Hu et. al. PRL 87, 148101 (2001)

Hu et. al. Acta. Cryst. D60, 621(2004)

Tomography

High Throughput 3D Tomography Tools

- Efficient sample handling & precision alignment
- Local and remote beam line operation
- Fast computational system for data analysis

sample changer robot

QuickTime™ and a
YUV420 codec decompressor
are needed to see this picture.

720 projections 1024x1024.
Total collection time:

    Si(111) mono   200 ~ 700 min
    Multilayer mono   5 ~   30 min

March 04 run (2 weeks) data:

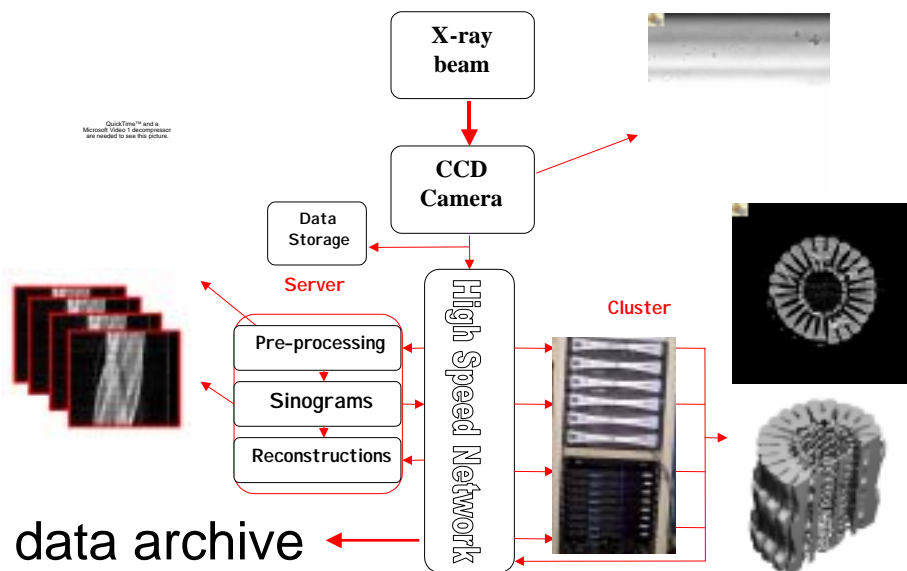|	
Acquired Images	388,141
Processed Data (GB)	5,081
Shifts	49
GU groups	5

Data throughput: 3.4 GB/min

Data Collection and Analysis

Reconstruction time:
50s (512x512); 3 min (1024x1024)

Remote operation

Developed by B. Tieman, APS/ANL

Remote control of the experiment
- Automatic data collection, preprocessing and reconstruction
- Electronic log

All users leaves the APS with fully analyzed data !!

Concrete

Sample 12.5 x 12.5 mm^2, 35 keV,900 proj, 4 hours.
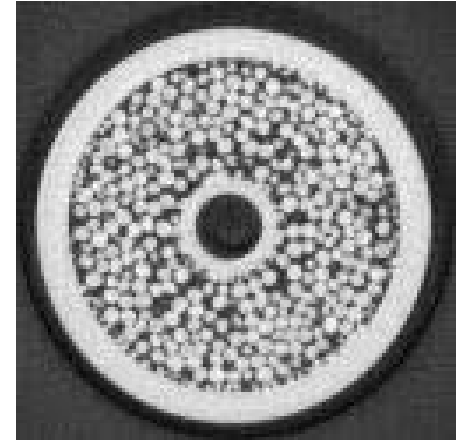Done at 5-BM DND-CAT.

Fig. 13. Reconstructed volume at 92% of peak load and 13% of peak load (post-peak) for sand aggregate

Title: Measuring three-dimensional damage in concrete under compression
Author(s): Lawler JS, Keane DT, Shah SP
Source: ACI MATERIALS JOURNAL 98 (6): 465-475 NOV-DEC 2001

Denis T. Keane • dtkeane@northwestern.edu • DND-CAT/Materials Science and Eng. Dept. • Northwestern University

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COMPLEX SYSTEMS - Sand

Signatures of granular microstructure in dense shear flows, D. M. Mueth et. al., Nature 406, 385-389, 2000. (13-ID)

Granule-by-granule reconstruction of a sandpile from x-ray microtomography data, G. T. Seidler, et. al., Phys. Rev. E 62, 8175-8181, 2000. (20-ID)

Microtomography Studies of Highly Explosive Materials

C. Bronkhorst – Los Alamos National Laboratory

- Development of a constitutive model of PBX 9501 (HE)
- Material modeling for direct numerical simulation for material and impact on explosion properties
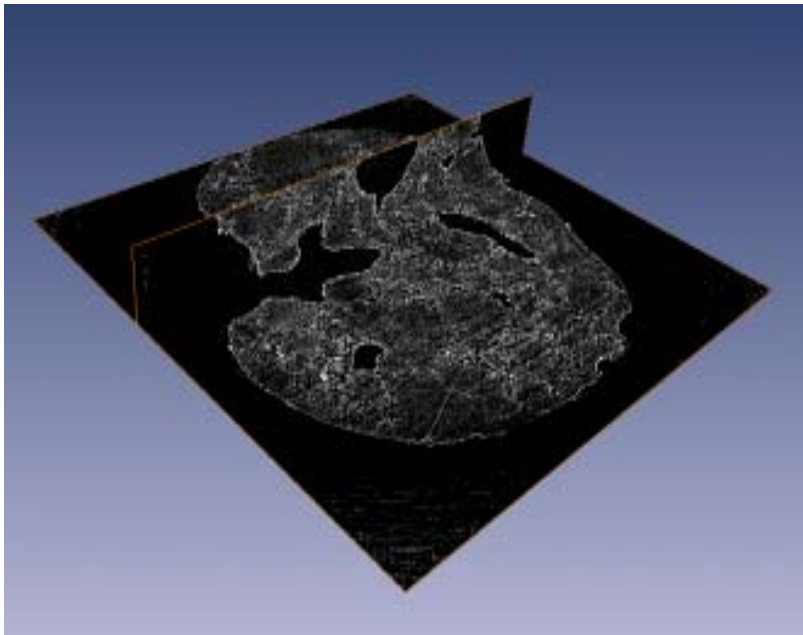
QuickTime™ and a
YUV420 codec decompressor
are needed to see this picture.

Biological Applications

• Studies of biomechanical strength and toughness through biomineralization.
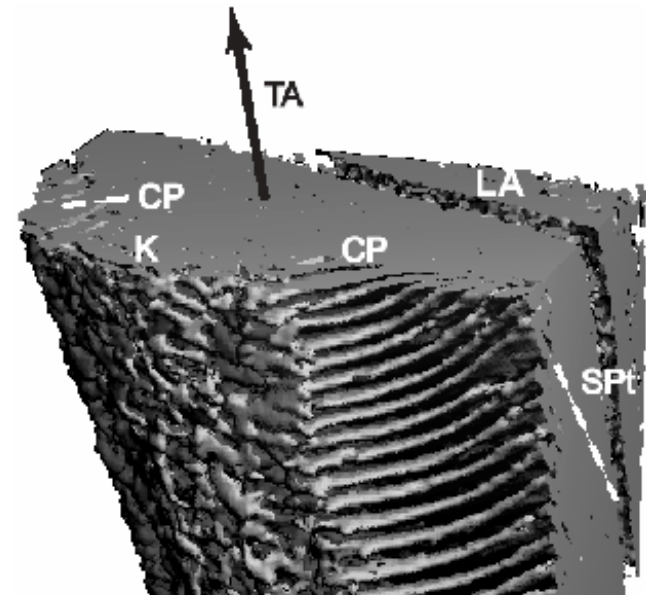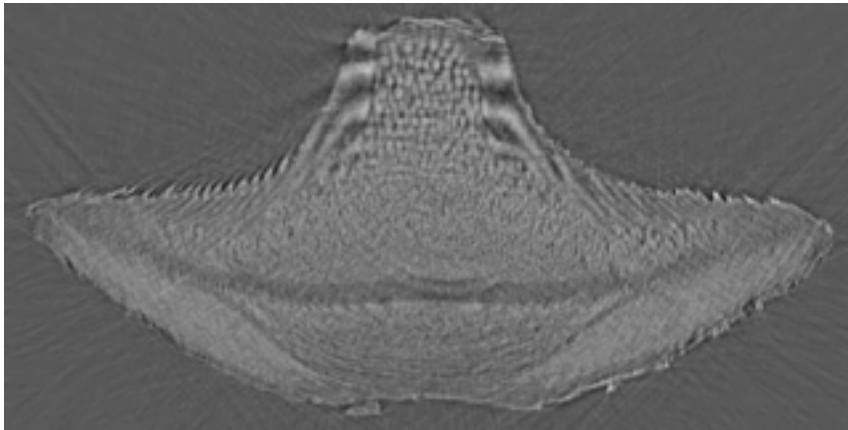
Sea urchin tooth cross section (left) and 3D rendering (right). In this rendering, the fragment is viewed from the base of the keel K. The horizontal cross section on the left is ~1.2 mm wide *(courtesy of Stuart Stock, Northwestern Univiversity).*
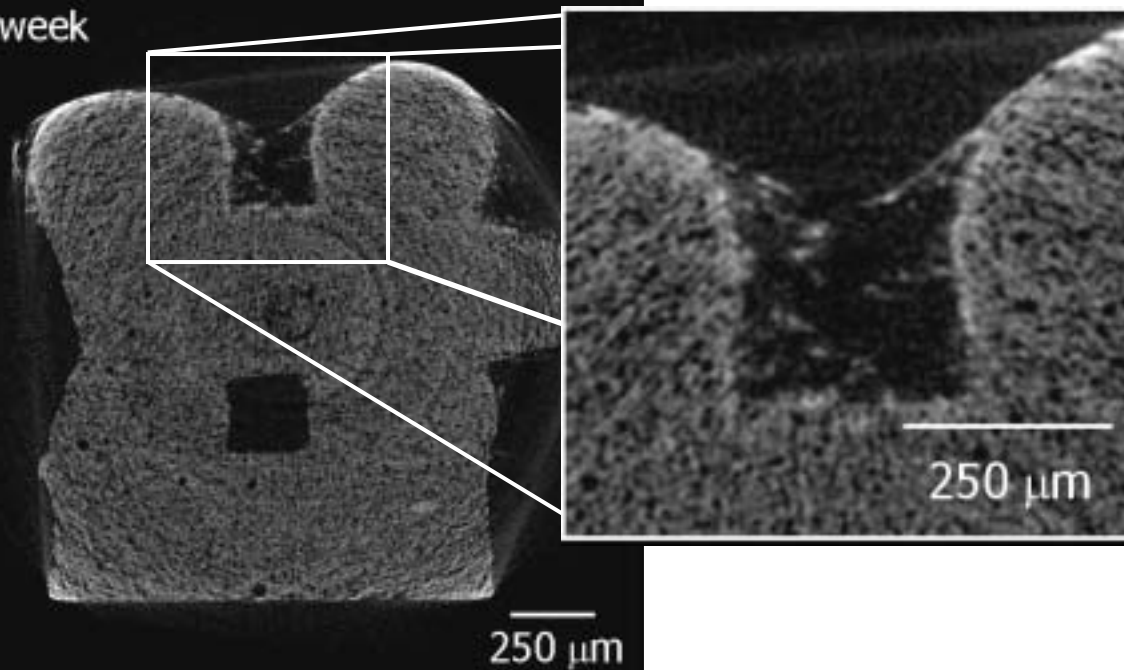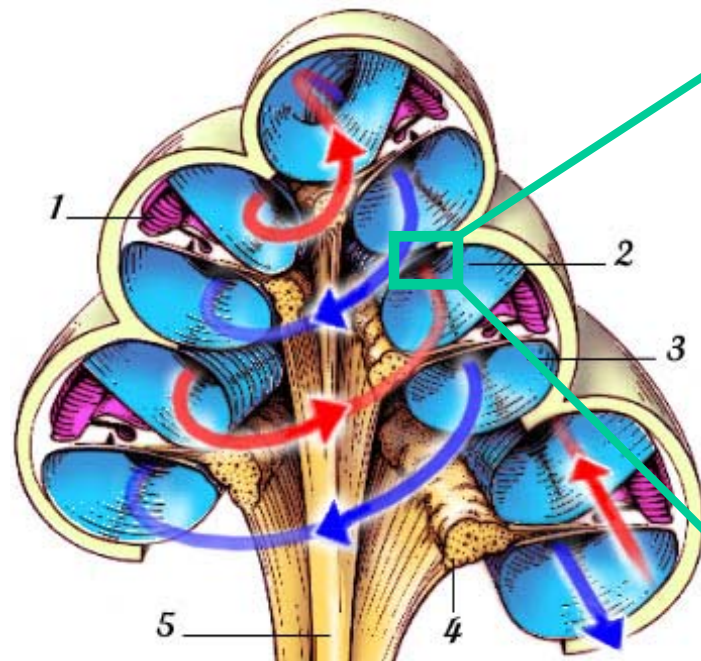
See talk by Stock (after dinner, Monday)
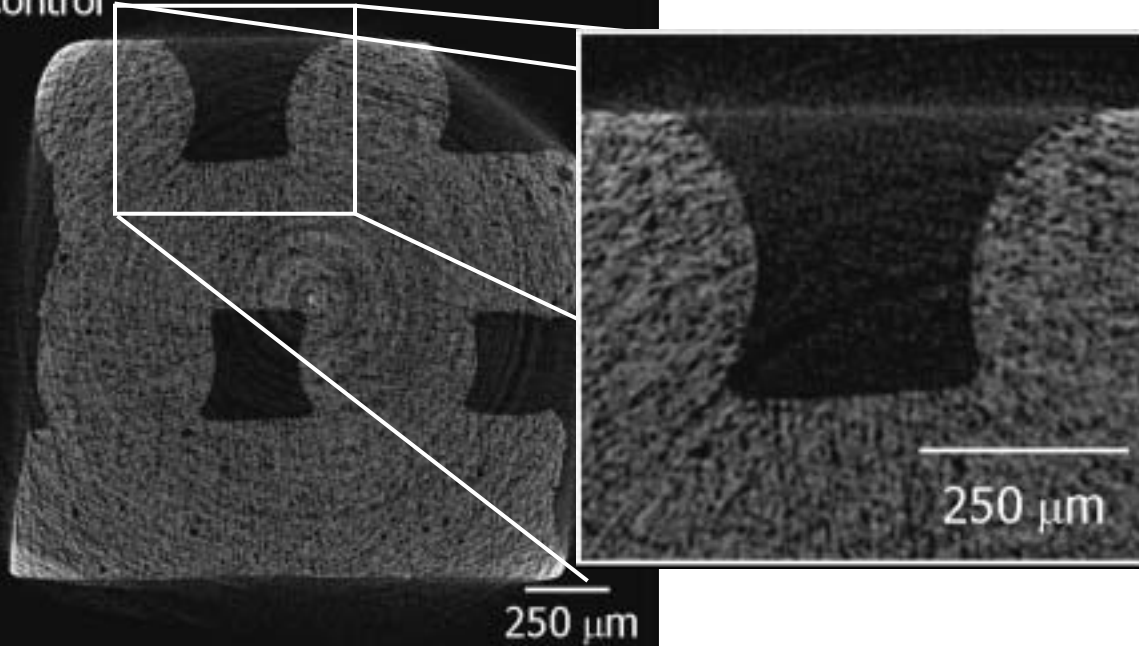
Bone tissue on porous hydroxyapatite (HA) scaffolds for tissue engineering applications.

For this study, tissue was cultured on HA scaffolds for 2 weeks and then post-fixed with OsO_4 to create an attenuation contrast between tissue and scaffold.

courtesy of Abbey Wojtowicz, University of Illinois at Urbana-Champaign

200 micron

- Better understanding of hearing process
- Dynamic study

C. Rau, 34-ID UNICAT

The high flux density naturally leads to time-resolved work…..

High flux, phase-enhanced contrast -> a new way of looking at small animal physiology

- good survivorship - hours

- good contrast for trachea

- satisfactory mounting - no evidence of stress

QuickTime™ and a
Sorenson Video 3 decompressor
are needed to see this picture.

Related avenues:

- complex mouth motions

- digestion (Steve Cook)

FOV=3x2 mm^2, 25 keV, 30Hz

Pro-thorax of beetle

See talk by Westneat (A2), Harrison (B2) & Cook (C2)

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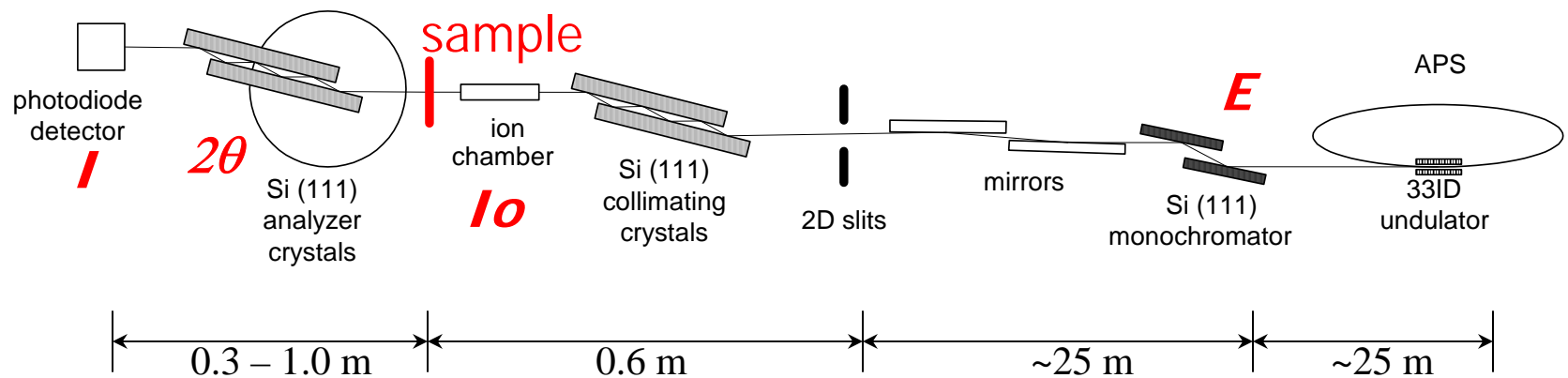Even higher flux + phase-enhanced contrast -> Micro-second imaging

FOV = 1x1 mm^2, filtered WB, stainless steel fuel injector

QuickTime™ and a
DV/DVCPRO - NTSC decompressor
are needed to see this picture.

See talk by J. Wang (session IV)

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Ultra small angle x-ray scattering imaging

Ultra-small-angle X-ray scattering imaging

- Parameters:
  - $1 \times 10^{-4} < q < 1.0$ Å^{-1}
  - 7 keV $<$ photon energy < 17 keV
  - $\Delta q = 1 \times 10^{-4}$ Å^{-1}
- Sample volume 0.4 mm $\times 2.5$ mm $\times 0.1$ mm $= 0.1$ mm^3
- Can measure selected areas of the sample
- Can perform raster measurements to cover large regions
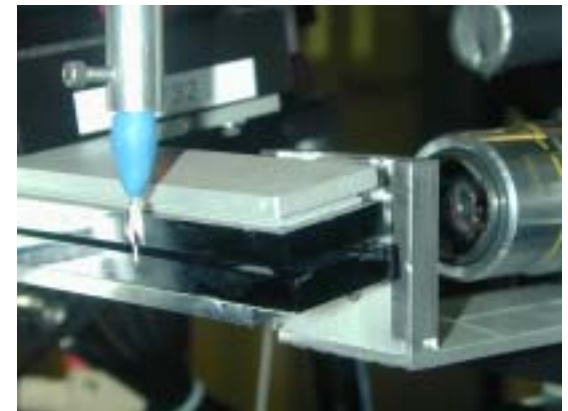- Opportunity to image the objects doing the scattering

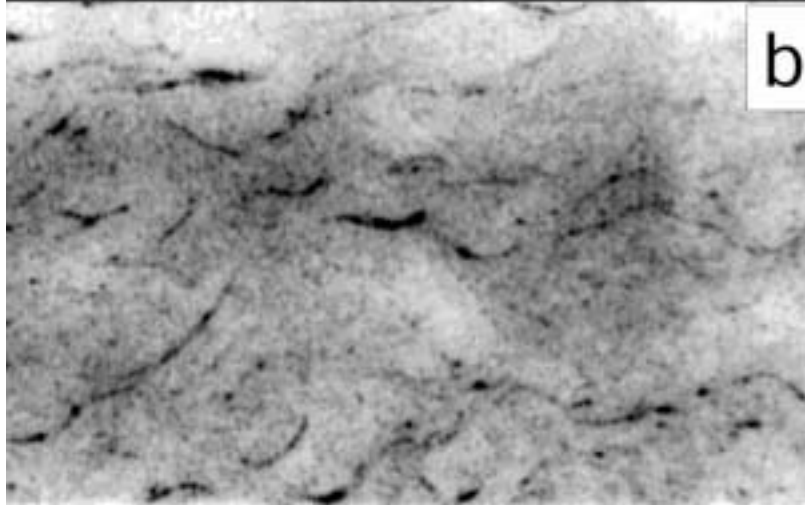See talk by Levine, session C-1

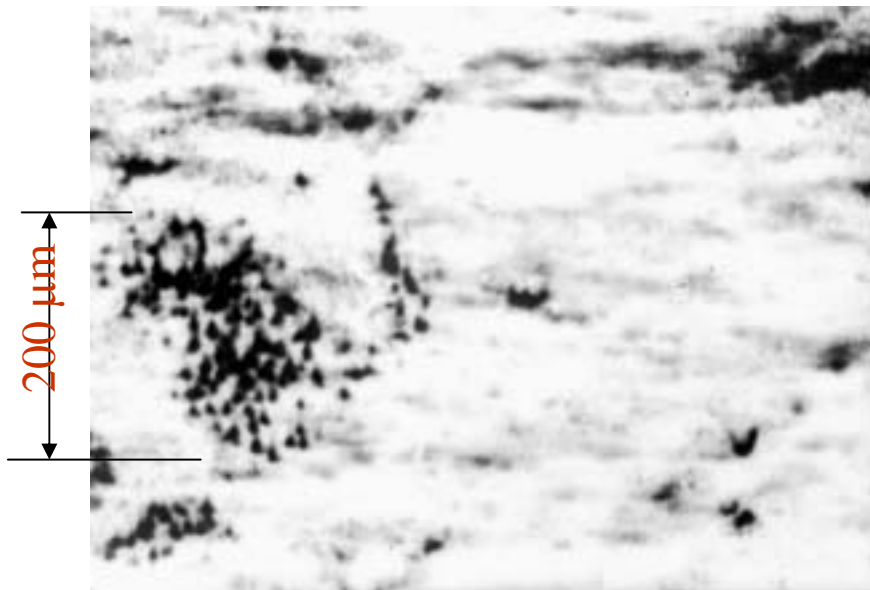# *Images of artificial tissue scaffold + cells*

q = 0.0 Å⁻¹
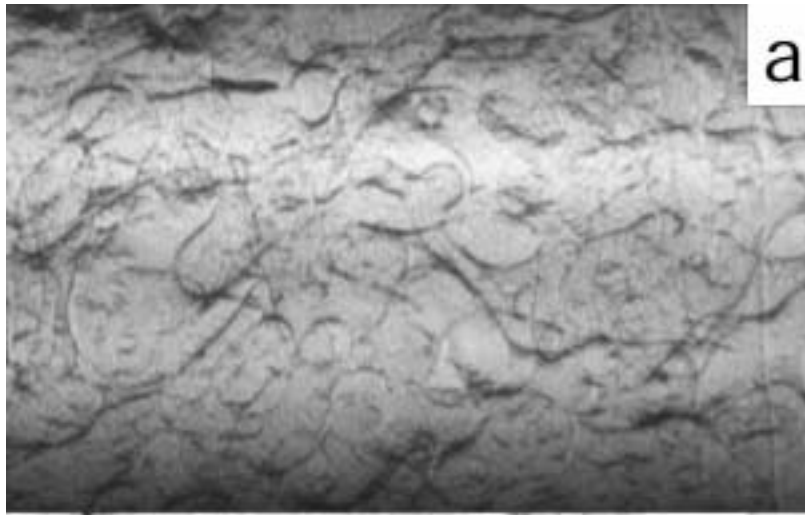
q = 0.0005 Å⁻¹

Sample, analyzer and detector

Porous poly-capralactone (PCL) with 50% porosity, cultured with osteoblasts for 28 days.

At low q, large (~ 12 µm) pores are visible

Crept, transverse
$q = 0.00013$ Å^{-1}

Resolution = 2.9 µm
E = 8.94 keV

Identical sample volumes

At larger q, dense regions of small
(< 3 µm) features are visible

Crept, transverse,
$q = 0.00075$ Å^{-1}

200 µm

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Perspectives

Sensitivity

20 µm

Spatial Resolution

x-ray

300 nm
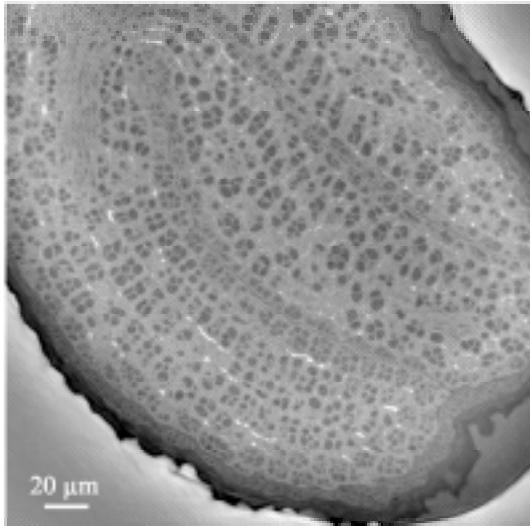
Dec 22, 1895. Mrs. Roentgen's hand

Time resolution

QuickTime™ and a
DV/DVCPRO - NTSC decompressor
are needed to see this picture.

Tomography

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Perspectives - techniques

- Combination imaging techniques

    - Phase sensitivity & tomography -> holotomography

    - Time resolution & phase sensitivity -> real time phase enhanced radiography

    -Ultimate imaging tool: Nanometer phase real time tomography???

- Combination x-ray techniques

    - Imaging & scattering

- Many optical techniques have yet to be realized in the x-ray regime

    - Fluid flow techniques

    - Tagged agents

- ???????

- Nanometer resolution related issues

  - Most samples are macroscopic ~ millimeter size.

  - Tomography at nanometer resolution would yield ~ 10^{18} bytes of data!

  - Even assuming computer progress, data mining would still be a major challenge

  --> Need to be able to 'zoom' into interesting area

  --> NEED LOCAL TOMOGRAPHY

Perspectives - applications

• Established x-ray imaging applications (Physics, Material Science, Medicine) will continue

• Real time applications - crack propagation, small animal physiology, functional imaging

• Emerging applications

     - Paleontology

     - Small animal physiology

     - Archaeology

     - Protein crystallography?

     - Nano-science?

     - ?????